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TAMPERE UNIVERSITY OF TECHNOLOGY

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**Comparison of Sintering Methods and Conductive
Adhesives for Interconnections in Inkjet-Printed Flexible
Electronics**



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Abstract

Increasing demands for flexibility and stretchability for electronic devices are driving the research for novel fabrication technologies. Inkjet-printing is one of these novel electronics fabrication techniques studied and developed globally in recent years and it has some interesting benefits over traditional lithography-based techniques, mainly its additive and digital nature. Traditional manufacturing methods are mature techniques and the processes are well defined and optimized for large scale manufacturing and inkjet-printing is not going to replace the lithography as such for large scale manufacturing. Inkjet-printing does, however, enable whole new ways of electronics fabrication, such as high part-to-part customization and 3D processability, which have previously been either very challenging or even impossible.

So far research has focused mainly on inkjet-printing itself and the jetting process is understood fairly well. However, at the moment printed semiconductor materials are far inferior to traditional semiconductor components and can not enable the same level of functionality or connectivity. Hybrid systems, combining the high performance of traditional semiconductor components and benefits of inkjet-printing, are studied as a solution for fabricating high performance devices with novel fabrication techniques. Hybrid systems require the ability to attach external components to the printed structures and this integration was chosen as one of the main topic for this thesis work as it had not been studied previously and the knowledge was required for developing inkjet-printing.

This thesis analyzes inkjet-printed hybrid systems and focuses on system level integration. The work is done on interconnections including both the sintering of metallic nanoparticles as well as external component interconnections and circuit board to circuit board connections. Sintering research is focused on alternative sintering methods to traditional thermal sintering and

evaluation of their usability in electronics fabrication. Electrically conductive adhesives are studied as the main method of forming external connection to components and to other circuit boards.

In the research related to this thesis alternative sintering methods were found to be suitable replacements for traditional thermal sintering with the advantages and disadvantages varying between different technologies. Laser and intense pulsed lighting were generally found to be the most promising techniques for inkjet-printed structures. External connections to traditional surface mounted components as well as other printed circuit boards were also successfully demonstrated in the related publications using electrically conductive adhesive materials. Both the electrical performance and long term reliability of the conductive adhesives were found to be inferior to solder-based interconnections but observations show that the difference is caused by the adhesive material itself, not by the use of inkjet-printing. Thus adhesives can be considered as a viable method for forming external interconnections on inkjet-printed structures.

Preface

The research discussed in this thesis was carried out at Tampere University of Technology, Department of Electronics and Communications Engineering, between 2010 and 2015 as well as at Friedrich Schiller University of Jena between 2010 and 2011.

The work was financially supported by Department of Electronics and Communications Engineering, Nokia Foundation, Tuula and Yrjö Neuvo Foundation, Ulla Tuominen Foundation, Finnish Science Foundation for Economics and Technology (KAUTE), Walter Ahlström Foundation and Finnish Funding Agency for Technology and Innovation (TEKES).

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Juha Niittynen

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List of publications

This thesis is based on the work presented in the following publications:

- [P1] J. Niittynen, E. Halonen, M. Mäntysalo, J. Perelaer, U.S. Schubert and D. Lupo. Conductivity and Adhesion Study of Plasma-sintered Nanoparticle Silver Ink. In *3rd Large-area, Organic and Printed Electronics Convention (LOPE-C)*, Frankfurt, Germany, 2011.
- [P2] J. Niittynen, M. Mäntysalo, D. Lupo, R. Abbel, J. Perelaer and U.S. Schubert. Comparison of photonic sintering of two inkjet-printed nanoparticle silver inks. In *4th Large-area, Organic and Printed Electronics Convention (LOPE-C)*, Munich, Germany, 2012.
- [P3] J. Niittynen, R. Abbel, M. Mäntysalo, J. Perelaer, U.S. Schubert and D. Lupo. Alternative Sintering Methods Compared to Conventional Thermal Sintering for Inkjet Printed Silver Nanoparticle Ink. *Thin Solid Films*, 2014, 556, 452-459.
- [P4] J. Niittynen and M. Mäntysalo. Characterisation of Laser Sintering of Copper Nanoparticle Ink by FEM and Experimental Testing. *IEEE Transactions on Components, Packaging and Manufacturing Technology*, 2014, 4, 2018-2025.
- [P5] J. Niittynen, E. Sowade, H. Kang, R.R. Baumann and M. Mäntysalo. Comparison of laser and intense pulsed light sintering (IPL) for inkjet-printed copper nanoparticle layers. *Scientific Reports*, 2015, 5, 8832.
- [P6] J. Niittynen, V. Pekkanen and M. Mäntysalo. Characterization of ICA-Attachment of SMD on Inkjet-Printed Substrates. In *IEEE 60th Electronic Components and Technology Conference (ECTC)*, Las Vegas, Nevada, USA, 2010.

- [P7] J. Niittynen, J. Kiilunen, J. Putaala, V. Pekkanen, M. Mäntysalo, H. Jantunen and D. Lupo. Reliability of ICA attachment of SMDs on inkjet-printed substrates. *Microelectronics Reliability*, 2012, 52, 2709-2715
- [P8] J. Niittynen, S. Koskinen, J. Kiilunen, J. Pippola, L. Frisk and M. Mäntysalo. Reliability of Flex-to-Flex Interconnections on Inkjet-Printed PCBs Using Electrically Conductive Adhesives. In *SMTA Pan Pacific Microelectronics Symposium*, Big Island, Hawaii, USA, 2014.

Author's Contribution

Publication 1, "Conductivity and Adhesion Study of Plasma-sintered Nanoparticle Silver Ink" is the author's contribution with help from co-authors. The author did the print trials, sintering and electrical measurement as well as wrote the manuscript with help from the co-authors. Co-author E.H. helped with the adhesion analysis.

Publication 2, "Comparison of photonic sintering of two inkjet-printed nanoparticle silver inks" is the author's contribution with help from co-authors. The author did the print trials, part of the sintering and electrical measurements as well as wrote the manuscript with help from the co-authors. Co-authors R.A. and J.P. helped with the sintering trials.

Publication 3, "Alternative Sintering Methods Compared to Conventional Thermal Sintering for Inkjet Printed Silver Nanoparticle Ink" is the author's contribution with help from co-authors. The author did the print trials, most of the sintering, result analysis and wrote the manuscript with help from the co-authors. Co-authors R.A. and J.P. helped with sintering, imaging and result analysis.

Publication 4, "Characterisation of Laser Sintering of Copper Nanoparticle Ink by FEM and Experimental Testing" is the author's contribution with help from the co-author. The author did the experimental print and sintering trials as well as FEM simulation. Result analysis and writing the manuscript were done by the author with the help from the co-author.

Publication 5, "Comparison of laser and intense pulsed light sintering (IPL) for inkjet-printed copper nanoparticle layers" is the author's contribution with help from the co-authors. The author did all the printing trials as well as the laser sintering trials. Co-authors E.S. and H.K. helped with the IPL

trials. The author also analyzed the results and wrote the manuscript with help from the co-authors.

Publication 6, "Characterization of ICA-Attachment of SMD on Inkjet-Printed Substrates" is the author's contribution with help from co-authors. The author carried out the print trials, component attachments and electrical tests as well as wrote the manuscript with help from the co-authors. Co-author V.P. helped with component attachment and result analysis.

Publication 7, "Reliability of ICA attachment of SMDs on inkjet-printed substrates" is the author's contribution with help from co-authors. The author carried out the print trials and component attachment and wrote the manuscript with help from the co-authors. Co-authors J.K. and J.P. helped with the cross-sectional imaging and structural analysis.

Publication 8, "Reliability of Flex-to-Flex Interconnections on Inkjet-Printed PCBs Electrically Conductive Adhesives" is the author's contribution with help from co-authors. The author did the print trials, component attachment, reliability testing and structural analysis as well as wrote the manuscript with help from the co-authors. Co-authors S.K., J.K. and J.P. helped with print trials, component attachment and reliability testing.

List of symbols and abbreviations

3D	Three Dimensional
ACA	Anisotropically Conductive Adhesive
Ag	Silver
CMOS	Complementary Metal-Oxide-Semiconductor
CTE	Coefficient of Thermal Expansion
Cu	Copper
DOD	Drop on Demand
DPI	Drops Per Inch
ECA	Electrically Conductive Adhesive
EMR	Electromagnetic Radiation
FEA	Finite Element Analysis
FEM	Finite Element Method
FIB	Focused Ion Beam
IC	Integrated Circuit
ICA	Isotropically Conductive Adhesive
IPL	Intense Pulsed Light
LASER	Light Amplification by Stimulated Emission of Radiation
PC	Polycarbonate
PCB	Printed Circuit Board

PEN	Polyethylene naphthalate
PET	Polyethylene terephthalate
PI	Polyimide
RF	Radio Frequency
RFID	Radio Frequency Identification
SEM	Scanning Electron Microscope
SMD	Surface Mounted Device
T _g	Glass transition temperature
VIA	Vertical Interconnect Access

Chapter 1

Introduction

In recent decades electronics research is mainly dictated by two topics; on one hand the semiconductor fabrication and on another the packaging technology. Since the development of transistor in the 1940's the transistor count and function-per-area ratio on a silicon chip has been steadily increasing, following a so called Moore's law, thus enabling ever faster processing speeds and smaller chip sizes. [1] Similarly, progress in packaging methods has enabled denser and more compact electronics assembly. Most important development steps for electronics packaging are surface mounted components, system-on-package and multichip modules as well as wafer level packaging solutions. [2]

While the semiconductor and packaging solutions have been developed, fairly little attention has been paid to the circuit board technology in the recent decades. However, now electronics industry is also looking at the circuit board technology for new innovations and possibilities of new types of devices and applications. Fabrication of electrical circuits on flexible and stretchable substrates enables devices with high adaptability to various forms and shapes. For example, new electronics manufacturing technologies have enabled stretchable devices, such as conformal temperature sensors for human skin and conformal energy harvesters working on the surface of human organs. [3, 4]

Printed electronics is one of the novel electronics fabrications to be studied and developed and it is predicted to grow into a significant business in its own right. [5] Printed electronics covers a wide array of different fabrication techniques with varying advantages and disadvantages. Different techniques

include screen, flexo, gravure and inkjet-printing among others. [6–8] Printed electronics enable processing of a large variety of materials and easy processing on flexible substrates and it has been used for fabrication of flexible displays, batteries, radio frequency identification tags (RFID), basic circuitry, antennas and component interconnections. [9–14]

Inkjet-printing is one of the interesting electronics manufacturing techniques, offering high productional flexibility and customization due to digital controlling and maskless fabrication. [15] Digital process control enables rapid prototyping and high product-to-product customization. Inkjet-printing is also a contact-free fabrication method and thus enables fabrication on a huge variety of materials and even on surfaces with 3D topography which would be challenging for most of the other fabrication techniques. [16–18]

1.1 Objective and scope of the thesis

Inkjet-printing and other novel printing methods have been extensively studied in recent years and several dissertations have focused on the printing processes themselves. [15, 19–23] Very little attention has, however, been paid to the system architecture: interconnections required to connect these printed structures to the outer world. This thesis work does not focus on jetting process itself, but on the next level of integration: sintering of nanoparticle inks and interconnections for external components and power and/or signal connections.

The main objective of this thesis was to form a better understanding of different methods which can be used to form interconnections to inkjet-printed structures. The main hypothesis was that a feasible and reliable method for fabricating interconnections on inkjet-printed structures can be fabricated. In this thesis interconnections related to inkjet-printed structures have been divided into two distinct categories: internal and external interconnections.

In the scope of this thesis internal interconnections refer to interconnections made between different printed components, such as antennae and energy storage as well as the sintering process of nanometallic inks themselves. Sintering of nanometallic inks is obviously fundamental to the use of inkjet-printing in electronics manufacturing, and the internal interconnections between printed components is also important when moving towards a higher

integration level and fabrication of functional systems instead of individual components.

External interconnections refer to interconnections between inkjet-printed circuit boards (PCB) and external components, such as traditional surface mounted devices (SMD) or silicon chips, as well as external connections to the outside world for the sake of power and/or signal connectivity. [14,24,25] Connections between several PCBs are also discussed.

The main reason for the use of external interconnections is the crucial need for external components in order to improve functional complexity. Printed semiconductor components, such as diodes and transistors, have already been fabricated but the size and performance is far from that required for today's consumer electronics. Semiconductor mobility and channel width ($\sim 10 \text{ cm}^2/(\text{V}\cdot\text{s})$ and $\sim 10 \mu\text{m}$, respectively) are both several orders of magnitude from those achievable on traditional silicon circuits ($\sim 1400 \text{ cm}^2/(\text{V}\cdot\text{s})$ and $\sim 10 \text{ nm}$). [26] Therefore traditional silicon-based components are used in so called hybrid designs, utilizing the high performance and reliability of complementary metal-oxide-semiconductor (CMOS) technology and the productional flexibility of novel printing techniques.

Figure 1.1 illustrates the different integration levels in electronics packaging. Level 0 includes wafer level packaging and all the interconnections made on the wafer surface. Level 1 covers integration of the silicon chips into a component package and in most cases also the required wiring to decrease the interconnection density in the following integration levels. Redistribution of the interconnections can be done by wire-bonding or by introducing an entire redistribution layer to the packaging. Level 2 includes component assembly on a PCB and thus covers the component-to-component interconnections. Level 3 integration covers the PCB-to-PCB interconnections and is relevant for larger multi-PCB assemblies.

The work related to this thesis is focused on integration levels 2 and 3; interconnections between components on a PCB and interconnections between two PCBs. Inkjet-printing can, however, be also used to combine integration levels 1 and 2 by connecting bare silicon chips on a PCB level. [18] Sintering of metal nanoparticle inks can also be considered to be a level 0 integration technique.

Some very basic electrical devices can be fabricated without signal or power connectors to the outside world but this greatly limits the level of function-

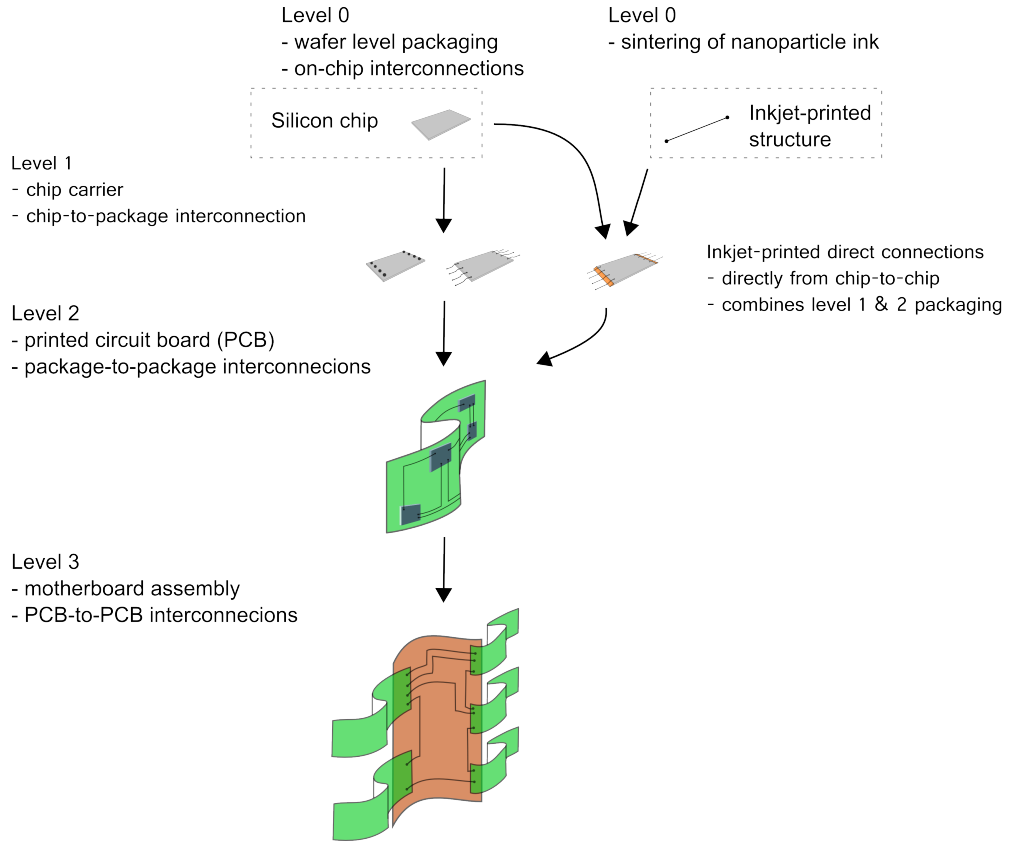


Figure 1.1: Illustration of the integration levels in electronics packaging.

ality available. For example low power sensors can be made without any physical connections to the outside world but the functionality is limited as the device has to rely on power transmitted via radio frequency (RF) transmission. Any data gathered by the sensor also has to be sent out via RF link using power absorbed from the incoming transmission.

Better understanding of the different interconnection techniques available for inkjet-printed structures is hoped to lower the technological risks for companies related to adopting novel manufacturing technologies in electronics manufacturing. Compatibility with existing manufacturing processes is an important factor in preparing inkjet-printing for large scale electronics manufacturing as it reduces the costs of adopting new techniques. The hope is also for the ground work done in this thesis to serve as a foundation for further research in the field.

1.2 Structure of the thesis

The thesis consists of an introduction and eight publications which present the main scientific results. The introduction is divided into five chapters which provide background for the publications. Chapter 1 discusses the general trends in electronics manufacturing and challenges the current fabrication technologies have in novel trends. Chapter 2 describes the fundamentals of using inkjet-printing for fabrication of electrical interconnections. Chapter 3 discusses the different available sintering techniques for metallic nanoparticle inks. Chapter 4 describes the methods for external interconnections and their long-term reliability testing. The final chapter summarizes the most important results for the entire thesis work as well as individually for each publication. The publications are appended to the end of the thesis.

Publication 1 investigates the plasma sintering of silver nanoparticle ink. Achieved conductivity was used as a measure of electrical performance and adhesion between the printed structure and the substrate as a measure of mechanical performance.

Publication 2 studied the intense pulsed light (IPL) sintering of two slightly different silver nanoparticle inks. The analysis is based on electrical performance, microstructure analysis and mechanical performance. Electrical conductivity and adhesion were used as measures of electrical and mechanical performance, respectively.

Publication 3 compares different alternative sintering methods available for replacing traditional thermal sintering for nanoparticle silver ink. Plasma, light amplification by stimulated emission of radiation (LASER) and IPL sintering are evaluated and compared to thermal sintering. Electrical and mechanical performance as well as microstructural analysis are used to analyze the different sintering methods. Electrical conductivity and adhesion between the printed structure and the substrate were used as measures of electrical and mechanical performance, respectively.

Publication 4 studies the laser sintering of copper nanoparticle ink based on finite element modeling (FEM) as well as experimental trials. The focus of the publication is to develop a computational model for evaluating the sintering temperature during the laser sintering process and estimate the accuracy of the model based on the experimental results.

Publication 5 compares laser and IPL sintering of nanoparticle copper ink. Electrical performance, evaluated based on conductivity measurements, and microstructural analysis are used as a basis for the comparative evaluation.

Publication 6 studies the electrical properties of external component interconnections on inkjet-printed PCBs utilizing electrically conductive adhesives (ECA) and traditional 0402 and 0201 sized SMDs. The study also includes flexible photolithography-fabricated circuit boards as reference cases as well as solder material for comparison with the ECA. The study seeks to identify the main factors affecting the electrical performance of component interconnections on inkjet-printed circuit boards.

Publication 7 focuses on long-term reliability of ECA-based interconnections on inkjet-printed PCB. The same test setup and samples were used as in Publication 6. Thermal cycling is used to induce accelerated environmental stresses on the tested devices.

Publication 8 investigates attaching two inkjet-printed PCBs together using ECAs. The focus is on electrical performance and long-term reliability. The study includes both isotropically conductive adhesives (ICA) and anisotropically conductive adhesives (ACA).

Chapter 2

Inkjet-printing of electrical interconnections

Printed electronics is researched as a possible alternative to traditional fabrication based on photolithography in electronics manufacturing. The term "printed electronics" itself covers a wide array of different fabrication techniques, such as gravure, flexographic, offset, inkjet-, aerosol jet and screen printing. These printing techniques differ greatly from each other, but the connecting feature is that all of these fabrication methods are additive techniques, as opposed to traditional fabrication methods which are mainly subtractive. An additive technique means a method of fabrication where material is only deposited on areas where it is needed, whereas in subtractive fabrication methods, such as traditional photolithography, the entire substrate is coated with material and unwanted material is later removed. Moving from subtractive to additive fabrication methods would reduce the amount of required energy as well as wasted material during the fabrication process. [27] For example in the traditional lithography process the majority of the original material is removed in the etching steps and therefore moving to no-waste additive manufacturing is a significant improvement. [28] Harmful chemicals and strong solvents are also used in etching processes. As the environmental consciousness of consumers increases, so does the pressure to move to environmentally friendly fabrication processes.

Inkjet-printing is one of the most promising printing techniques and has been studied extensively in recent years. [29–32] Its advantages, which it shares with aerosol jet, over traditional manufacturing techniques and other

printing techniques include a fully digital process control and contact free manufacturing. Inkjet- and aerosol-printing share several advantages as the two technologies are somewhat similar in their basic functions. The difference is that while inkjet-printing utilizes functional materials in liquid form, aerosol-printing utilizes vaporized materials in gas form. [33,34]

Digital manufacturing means that the fabrication process has no physical masks or stencils and the pattern to be fabricated can be changed simply by altering the digital control file. This gives significant advantage in rapid prototyping or manufacturing requiring a high level of part-to-part customization. A prime example of utilization of the part-to-part customization is an inline printfile modification tool which optically inspects the real positions and orientations of integrated circuit (IC) chips and modifies each printfile to match exactly the real life situations. [35]

Contactless manufacturing means that with inkjet-printing electrical patterns can be fabricated on surfaces without physical contact. This enables both fabrication on surfaces with three dimensional (3D) topography and substrate materials which are highly sensitive to physical contact. For example inkjet-printed patterns can be fabricated on substrate assemblies with SMD making 3D topography of over 1 mm in height. [17] Fabrication of electrical patterns on these kinds of substrate assemblies is extremely challenging with traditional, or even with most of the other printing, techniques.

2.1 Inkjet printers and printheads

There are two different basic working principles for inkjet-printing: continuous printing and drop-on-demand (DOD) printing. Continuous printing means that the printhead constantly produces ink droplets at a constant frequency and these droplets are directed either to the substrate to form the desired pattern or back to the printhead's circulation system using an electric field. [36] In DOD printing the ink droplets are only formed as needed and all of the formed droplets end up on the substrate. [36] DOD is more commonly used of the two technologies and has been used in all the publications and research work related to this thesis.

Printheads can also be divided into several categories depending on which droplet formation technique is used. The different techniques include piezo-

element, thermal, acoustic and electrostatic droplet formations. [36,37] Piezo-element based drop formation is the most commonly used as it is easiest to control and the most repeatable and reliable and compatible with most types of inks. [36] All publications and research work related to this thesis has been done with inkjet-printers using piezo-element based droplet formation.

Figure 2.1 illustrates the basic functionality of the piezo-element based DOD printhead. The computer controls the ink drop formation based on the input image file and drives the piezo-element in the printhead (purple element in Figure 2.1). The piezo-element expands according to the input signal and expansion forces ink out of the nozzle of the printhead forming an ink droplet. Ink droplets travel to the substrate and form a pattern on the substrate. On Figure 2.1 the relative motion of the printhead compared to the substrate is to the right.

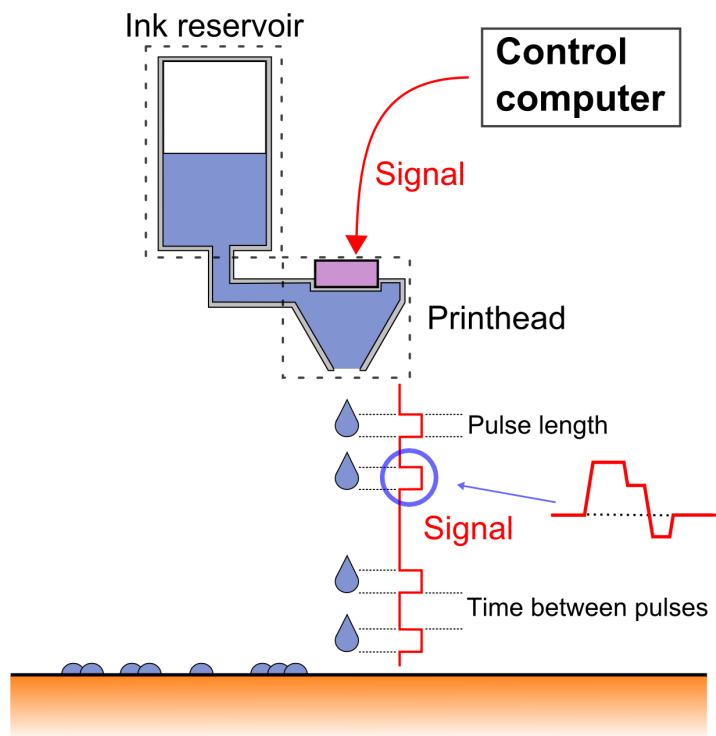


Figure 2.1: Illustration of the basics of a piezo-element based drop on demand printhead.

Figure 2.1 illustrates a single nozzle printing system but in most cases several nozzles are used simultaneously to increase the processing speed. The number of nozzles can go over 2000 in a single printhead and each nozzle is individu-

ally controlled by the software to make up the designed pattern. [38,39] Print-heads used in the experimental tests for to the publications are presented in Figure 2.2 and the larger prototype-scale inkjet-printer is presented in Figure 2.3. Smaller printhead on the left is the one used in Dimatix DMP-2831 and the one on the right is the Spectra SE-128 which is used in the iTi XY 2.0 MDS inkjet-printer made by Imaging Technology International (see Figure 2.3). Both printheads are made by Fujifilm Dimatix.

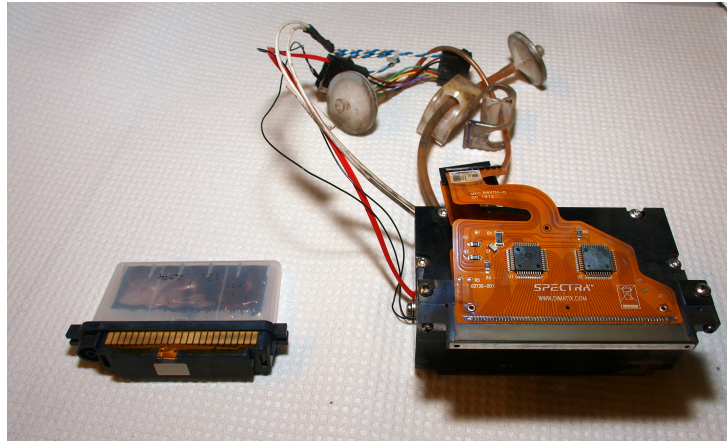


Figure 2.2: Printheads used in the experimental tests.

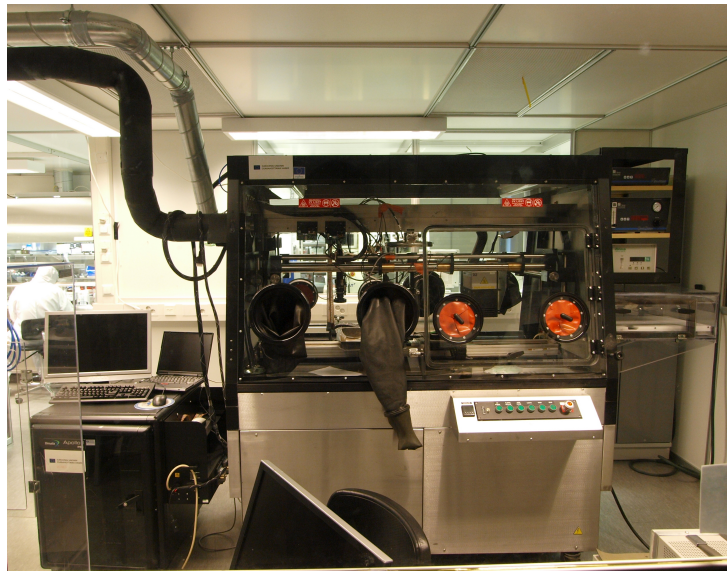


Figure 2.3: Prototype-scale inkjet-printer used in the experimental studies.

2.2 Materials

Materials needed for fabricating a electrical device by inkjet-printing include functional materials, such as metal nanoparticle inks, as well as substrate materials to act as a platform and offer mechanical support. In addition to functional materials themselves, other materials, such as cleaning and surface treatment chemicals, are used during the process.

2.2.1 Inks

Inkjet-printed materials used in electronics manufacturing can basically be divided into five distinct categories: conductive, dielectric, semiconductive, magnetic and piezo-electrical materials. Other uses for inkjet, besides the obvious graphical industry, can be found for example in biology. High control precision and repeatability of the inkjet-printing enable accurate dispensation of cell cultivation. [40, 41] However, this thesis only covers the electrically conductive inks.

Conductive inks are most commonly used for fabrication of electrical interconnections but even conductive inks include large variation. Metallic nanoparticle based inks are the most commonly used in inkjet-printing of electrical patterns but other options include metal complexes, salts, organo-metallic precursors and carbon nanotube based inks. [42–44] Metal nanoparticle inks are used in the research and publications related to this thesis due to their easy processability and good conductivity. Metal salts cannot provide as good conductivity as nanoparticle inks and their use might require complex chemical treatments. [45] Organo-metallic inks provide conductivities comparable to nanoparticle inks, around 50% of that of bulk material, but the low metallic loading makes their use challenging. [46]

In addition to functional substances, inkjet-printing inks include a liquid vehicle (water or alcohol solvent) to make the solution printable and a varying amount of additives meant to modify the printing process, ink-substrate interaction and/or post-processing behavior. [42] Ink composition is a delicate chemistry due to varying processing conditions and possible ink component interactions.

2.2.2 Substrate materials

Substrate materials most commonly used in inkjet-based electronics manufacturing include polymer foils, such as polyimide (PI), polyethylene naphthalate (PEN), polyethylene terephthalate (PET) and polycarbonate (PC), epoxy-based materials for rigid assemblies or ceramic materials for RF applications. [14,24,47] In most cases the substrate choice is affected by flexibility requirements of the application and the processing temperatures of the electronics fabrication technique to be used. [48]

Stretchable applications require use of stretchable substrates with highly repeatable and reversible elastic deformation characteristics. Most common substrate materials for stretchable applications include polyurethane and silicon based compounds. [3,49]

2.3 Pre-processing

Pre-processing for inkjet-printing includes the various substrate and surface modification processes available to modify the printing conditions. Cleaning the substrate of any contaminants is the most important part of the pre-processing. The main point is to remove any dust and grease from the substrate as they will otherwise harm either the print quality or the adhesion of the final product. The most common cleaning technique is using an alcohol-based cleaning solvent, isopropanol being one of the most common. Other cleaning methods include UV-ozone, plasma and corona treatments depending on the type of contamination and the used substrate material.

Next to cleaning, surface energy is one of the most important factors to be modified in the pre-processing step as it affects the spreading on the printed ink on the surface and therefore also ultimately dictates the resolution and patterning accuracy. Surface energy can be modified by plasma, ionization or chemical treatment. [50,51] Selective surface modification tools can be used to create hydrophobic and hydrophilic areas on the substrate and thus prevent or promote wetting and control the pattern formation. [50] Hydrophobic/philic surface processing can also be used to create channels and control liquids using capillary forces. [52,53]

Pre-processing may also include treating the ink to better suit the printing, such as degassing the ink to remove the air from the ink. Ink bubbles inside the ink can disturb the printing and clog the nozzles or cause open circuits and/or short circuits in the pattern.

2.4 Printing

Print processing includes the ink jetting and drying of ink on the substrate immediately after the jetting. Print process parameters have a significant effect on the printing quality as well as jetting reliability and repeatability. The most important jetting parameters when using piezo-element based droplet formation are jetting waveform, jetting voltage, ink temperature and pressure conditions on the printhead. [54, 55] These parameters dictate the ink droplet size, shape and speed upon jetting and therefore also the printing quality. These variables have to be mapped separately for each ink/printhead combination and in some cases even for each individual printhead due to part-to-part variation.

The droplet size can also be altered dynamically during the printing by using the so called "greyscale" mode. [39] In the "greyscale" method the print pattern can be designed in greyscale format instead of a binary bitmap and the different scales of grey can be assigned for different printing parameters. This approach can be applied to cases in which varying layer thickness is wanted inside a single device. [13] Varying layer thickness can also be achieved with multiple printing runs, but the "greyscale" method offers a simple alternative as it only requires one print run.

The accuracy of inkjet-printing is affected by the printing height (distance between the printhead and the substrate), stability and speed of the ejected droplets as well as the printing speed (relative speed by which the printhead and print plate move to each other). As inkjet-printing is a fully digitally controlled process, placement errors caused by constant variables, such as printing height and speed, can be taken into account in the print file and thus eliminated. [56]

After jetting the ink hits the substrate and the behavior of the ink on the substrate is dictated by the material parameters of both the ink and the substrate surface. The main factors include viscosity of the ink as well as

surface roughness, surface energy and temperature of the substrate. In most cases the viscosity of the inkjet inks are highly temperature dependent and can therefore be controlled by heating the ink before jetting. Surface energy modifications can be made in the pre-processing step.

Most inkjet-printers are equipped with a heated print plate and the elevated substrate temperature is commonly used in order to control the spreading of the printed ink. Heating may, however, contribute to the "coffee ring"-effect which is due to the material flow inside the ink during solvent evaporation. [57] The effect of the substrate temperature on the "coffee ring"-effect depends on the ink as different solvents behave differently. [58, 59] The "coffee ring"-effect can be minimized or even eliminated completely by using two solvent mixtures with different boiling points. [60]

Figure 2.4 illustrates the "coffee ring" effect on a sintered structure. The image has been taken with Wyko NT1100 optical profilometer which is based on white light interferometry. The size of the area imaged is 2.4 mm by 1.9 mm and the width of the printed conductor line is about 320 μm .

Another method for controlling the drying and therefore the spreading of the ink is the substrate surface energy modification prior to printing or modifying the used print file to maximize drying time for each droplet by changing the order of droplet deposition. This can be done for example by splitting the image into several images or by introducing additional rows into the image and thus increasing the resolution but keeping the amount of ink the same. [14, 24]

2.5 Post-processing

Post-processing includes all processes needed to turn the printed ink into the final functional structure. Required post-processing steps depend on the type of ink in question as different functional materials demand different post-processing techniques. The most common post-processing is thermal heating. The main purpose of post-processing is to fuse the printed material into a more uniform structure and/or accelerate the process, for example cross-linking of polymers. [42]

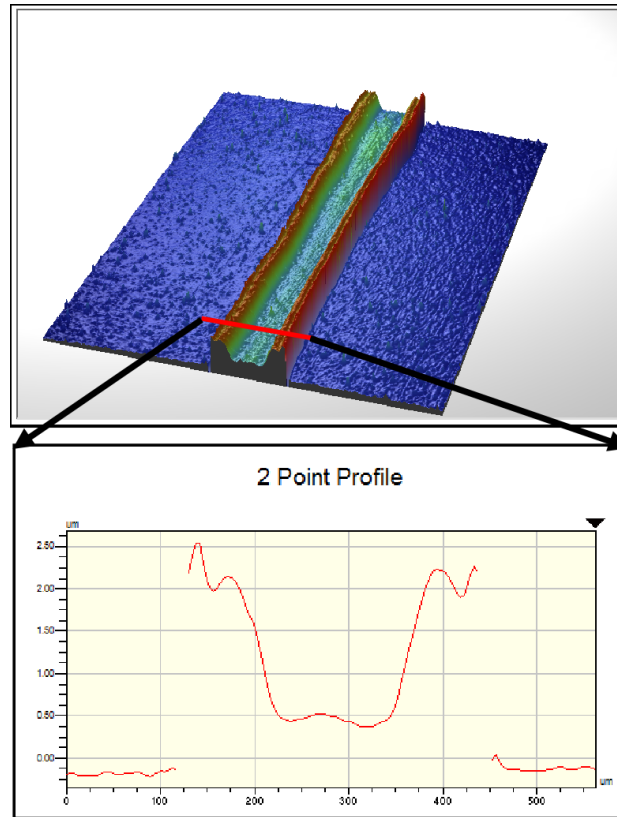


Figure 2.4: Illustration showing the "coffee ring" effect on a sintered structure.

Metal nanoparticle inks include number of small metal particles coated with a thin polymer layer used to prevent particle agglomeration before printing and ensure the printability of the ink. [42] Post-processing of metal nanoparticle inks is required to first remove the polymer shell of the metal particles before the actual sintering of the nanoparticles can occur. Sintering of metal nanoparticle inks is discussed in more detail in the Chapter 3.

Other needed post-processing steps may require applying various protective coatings on the printed structures. Protective polymer coatings can be used to enhance long-term reliability, especially in high humidity or high salt concentration conditions. [24,61]

Chapter 3

Sintering of metal nanoparticles for internal interconnections

An important requirement for microelectronic devices is that they all need conductive structures, which makes metals obvious choices due to their superior conductivity compared to organic materials. Electrical conductivities in the range of 100 S/cm have been achieved with printed conductive polymers, but that still leaves them several orders of magnitude below metal nanoparticles. [62] Inkjet-printing of metal containing precursor materials has been widely accepted as a suitable processing alternative for the fabrication of contacts and interconnects at relatively high speed that enables R2R production. The required sintering step after printing to render the precursor materials conductive, however, hampers the fast processing speed.

In order to gain conductivity from printed metal precursor inks, the organic layer that keeps the metal nanoparticles in solution should be removed, which forces the particles into close physical contact, followed by neck formation between the particles – a process known as Ostwald ripening or sintering. [63] Once the sintering process takes place a continuous percolating network is formed throughout the printed features, resulting in electrical conductivity. The organic layer is typically removed by heat and there are several different heat transfer methods available to be used.

It is important to evaluate the electrical performance of the printed and sintered features as it is necessary to achieve decent conductivity in electronic applications; low conductivity causes unnecessary power losses and a

decreased signal-to-noise-ratio throughout the device due to increased resistances, resulting in excess heating and possibly reliability issues may occur. Furthermore, low conductivity can disturb high frequency conductor lines and time-critical signal lines. [13, 64, 65]

Secondly, it is important to evaluate mechanical performance as it directly relates to reliability of the printed structures. Poor mechanical properties lead to decreased reliability and shortened lifetime, in particular in dynamic and flexible applications where mechanical stresses are more prominent than in static circuit boards. Ability to endure mechanical stresses caused by the flexible action is very important as flexible devices are considered as one of the most promising applications for printable electronics.

Electrical performance of the different alternative sintering techniques have been tested and studied quite thoroughly but so far the mechanical performance and thus reliability has not been studied nearly as much even though it is at least as important a factor in electronics manufacturing as electrical performance.

3.1 Thermal sintering

Traditional convectional heating with an oven is the most common sintering method. Most of the inks require high sintering temperature which makes then incompatible with cost-effective polymer foils, such as PET or PC, which have relatively low Tg. [66] As an alternative, more expensive polymer substrates, such as PI or polyarylate, can be used, but this obviously hampers the cost-efficient production process. [67] Thermal sintering is also a slow sintering process, taking typically from 30 to 60 minutes and is therefore poorly suited for large scale R2R manufacturing. [P3] [68]

Thermal sintering enables high conductivity and very good adhesion for silver nanoparticle inks. [P3] [68] However, the high processing temperature and long sintering time mean that alternative sintering techniques are studied and developed in order to make inkjet-printing better suited for large-scale electronics manufacturing.

3.2 Alternative sintering techniques

Several research groups have studied different alternative sintering methods in order to decrease both the sintering temperature and time. Instead of heating the complete sample, more selective sintering techniques are being studied, including laser sintering, low pressure argon plasma exposure, microwave radiation, electrical and IPL sintering. [69–73] These techniques have been successfully used to achieve electrical conductivities suitable for electronics applications.

Different sintering techniques are also studied in the publications related to this thesis. [P1-P5] The main focus of the sintering development is on electrical and mechanical performance. Plasma, laser and IPL sintering are discussed in more detail as these are the techniques analyzed in the research and the publications related to this thesis.

3.2.1 Plasma sintering

Plasma sintering utilizes a plasma flow to get rid of the dispersion agent protecting the nanoparticles and thus enabling the sintering process and formation of electrically conductive structures. [70] Plasma sintering also enables inkjet manufacturing on low cost thermally sensitive substrates such as paper, PET and PEN due to the low processing temperature of the plasma treatment. [71, 74]

Technically plasma sintering is based on plasma etching of polymer shell of the nanoparticle. The high surface-to-volume ratio of metal nanoparticles enables the sintering, and therefore formation of conductive features, even at low temperatures once the protective layer is removed. The temperature does, however, affect the achievable conductivity. Due to the low processing temperature of plasma sintering the achievable electrical conductivity is relatively low. [P1, P3]

Plasma sintering also suffers from a strong top-to-bottom behavior, meaning that sintering progresses from top of the printed structure towards the substrate and easily leaves the bottom layer unsintered. This is problematic as it also means that the adhesion between the printed structure and the substrate is insufficient for electronics applications. [P1, P3] [70] As plasma sintering

relies on plasma etching and as most of the printed electronics is done on plastic substrates, etching affects the substrate materials as well. [74, 75]

3.2.2 Laser sintering

The laser sintering technique utilizes laser radiation to get rid of the dispersion agent protecting the nanoparticles and to sinter the revealed nanoparticles together to form conductive structures. [69]

Laser sintering has been successfully used to sinter both silver and copper nanoparticles. [76, 77] Compatibility with copper nanoparticles makes laser and IPL sintering attractive alternatives as the inkjet-printing community is eager to change from silver materials to copper to reduce the manufacturing costs, but copper nanoparticles are significantly more challenging to sinter than silver nanoparticles due to the oxidation sensitivity of copper.

Copper nanoparticles are very sensitive to oxidation and therefore sintering is usually done under a protective atmosphere or a localized and directed gas flow, usually nitrogen, to prevent contact with oxygen. [77–79] Laser sintering, however, is rapid enough a process for oxide not to be formed between the copper particles and sintering can be successfully done in ambient conditions. [P4, P5] [80] A high speed of sintering may also cause problems as the rapid evaporation of solvent and/or dispersion agent material seems to be able to severely damage the microstructure of the printed structure. [P3]

Laser sintering differs from other sintering techniques by being a highly localized sintering method whereas other methods mainly affect larger areas at once. The size of the laser spot of the laser sintering equipment used in the publications related to this thesis was 1.1 mm by 400 μm whereas other sintering methods studied in the publications P1-P5 can sinter A4 sized sheets at once.

This means that sintering larger structures with laser requires scanning to cover the entire area. The principle of the scanning function of laser sintering is presented in Figure 3.1. In Figure 3.1 the darker red area represents the laser spot, the lighter red are the already sintered area and the black dashed line the travel path of the laser spot.

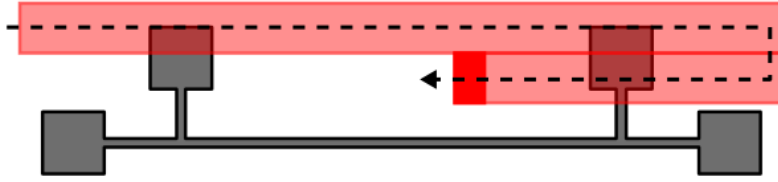


Figure 3.1: Illustration of the operation principle of laser sintering.

Due to the scanning function the processing time of laser sintering depends on the structure size and geometry whereas for other sintering methods the processing time is practically constant regardless of the structure size. Scanning speeds ranging from 50 mm/s to 200 mm/s were used in the publications related to this thesis, but speeds from 1 mm/s to 1 m/s have been successfully tested. The scanning speed and optical power have to be adjusted in combination to optimize the sintering conditions. [81] An alternative way is to use a larger matrix of laser diodes to cover a larger area at once. The custom-built laser-sintering setup used in the experimental tests for the publications P3, P4 and P5 is presented in Figure 3.2 with the exception of a slightly different lens setup.

Localized sintering enables a unique level of sintering control due to real-time digital control of both laser spot location and the optical power of the laser unit. It is possible to use a similar "greyscale" image file to set up different laser sintering powers on different areas of the printed structure by changing the optical power of the laser unit. This enables altering sintering power according to feature size and optimize the sintering condition to suit the entire structure instead of compromising between different feature sizes. Many patterns have a mixture of different sized features both in the X-Y direction and layer thickness-wise. [13, 14]

Adaptive optical power adjustment based on a pattern would also combine the benefits of digitally controlled deposition and post-treatment processes: inkjet-printing and laser sintering, respectively. This combination would further increase the potential of using inkjet-printing as a rapid prototyping tool benefiting from a high level of customization and accurate controlling as well as a complete lack of physical masks due to fully digital fabrication.

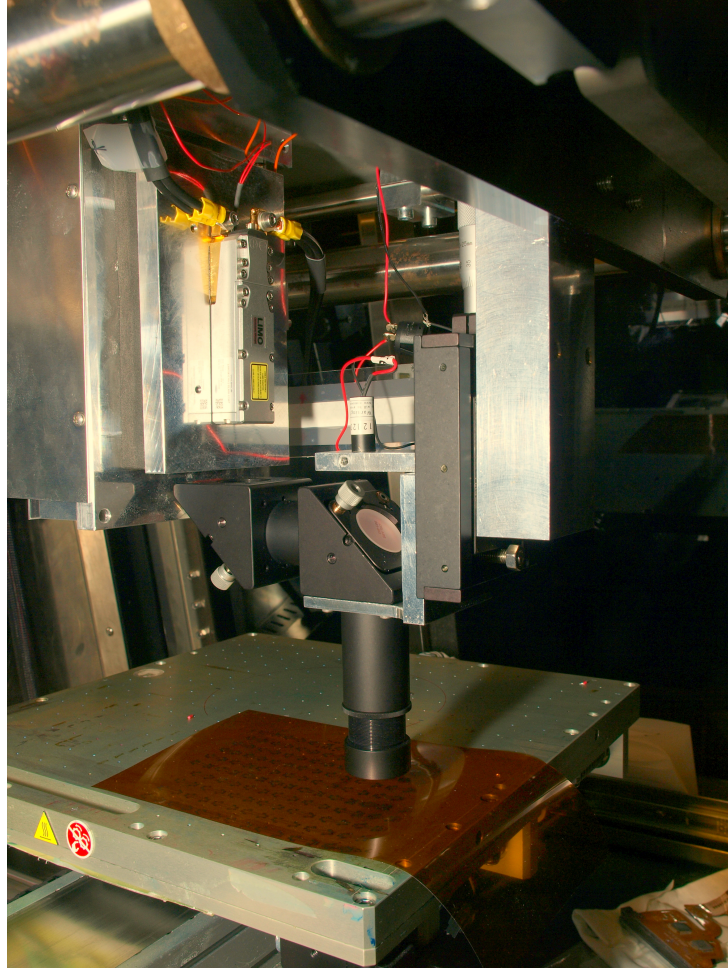


Figure 3.2: The laser-sintering setup used in the sintering experiments.

Modeling of laser sintering

Thermal analysis of laser sintering was modeled with the Finite Element Method (FEM) in order to analyze and better understand the thermal behavior of the printed structures during laser sintering. The main focus is on time response of the temperature and thermal dissipation in the printed structure during and after the laser spot has passed the specific area.

Challenges involved in the modeling of laser sintering include specifying the material properties and structure geometries. Many of the material parameters, especially those of inkjet inks, are not shared by the material manufacturers and estimations have to be made based on limited knowledge.

[P4] Both the geometry, especially vertical height, and the material parameters of the printed structure also change during the sintering process as the nanoparticles form a denser microstructure. For example thermal conductivity is increased as sintering progresses.

Figure 3.3 illustrates thermal distribution in a copper trace. The left side of the image includes already sintered copper while the right side has unsintered copper ink, and the higher thermal conductivity of the sintered copper mean that more thermal energy is conducted towards the already printed structure than towards the unsintered ink. Therefore sintered areas, especially large parallel plane capacitor and ground planes, basically work as a heat spreader and thus affect the sintering temperature.

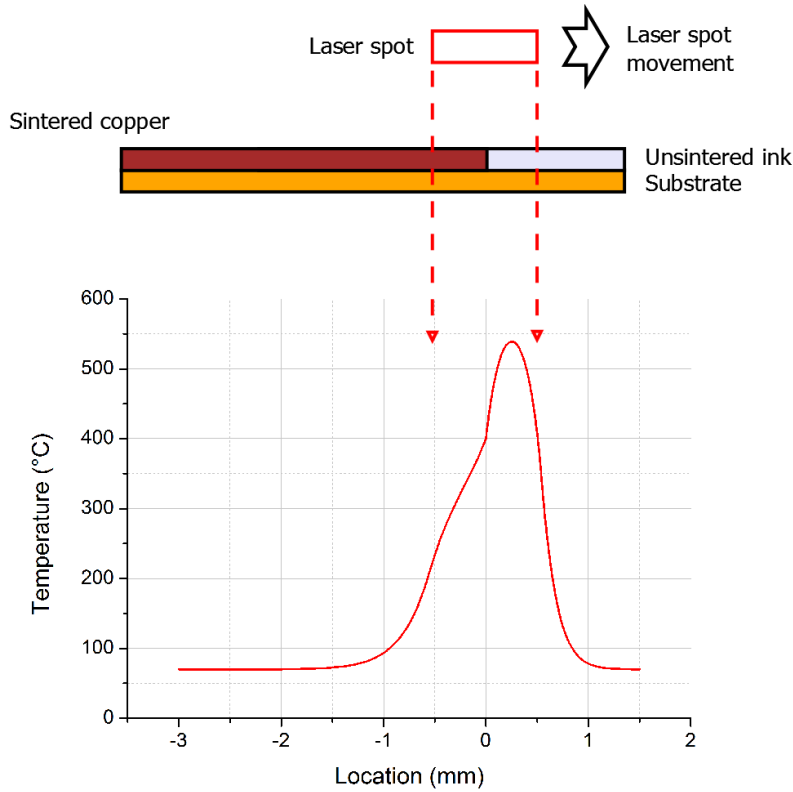


Figure 3.3: Illustration of the thermal distribution during laser sintering of a copper conductive trace. [P4]

Modeling of laser sintering can be used to estimate the sintering temperatures corresponding to specific sintering process parameters and therefore to calculate the optimal optical power for each pattern geometry. In an optimal

case the FEM model could be used to adjust the power level on the fly in the "greyscale" laser treatment.

Variable power sintering is therefore an important benefit as different feature sizes behave differently during sintering: larger areas, such as capacitor and ground planes, do not dissipate heat as effectively as thin conductor lines, such as those used in IC connections, and therefore sintering temperature may not be constant throughout a printed structure containing both large areas and thin lines. With other sintering techniques this may require some level of compromise: if the sintering power is set up according to the larger areas then the smaller thin lines may remain poorly sintered or even completely unsintered, and if the power is set up according to the thin lines the large areas may be destroyed due to overheating. [P5]

Figure 3.4 shows the modeled maximum reachable temperature on a printed conductor line as a function of the line width. The static analysis is based on a FEM analysis done with a $5\text{ }\mu\text{m}$ thick copper ink layer and 5 W sintering power over the laser spot of 1.1 mm by 0.4 mm. The modeling was done on top of a $125\text{ }\mu\text{m}$ thick polyimide substrate and all the material parameters and scaling factors were similar to the ones used in publication P4.

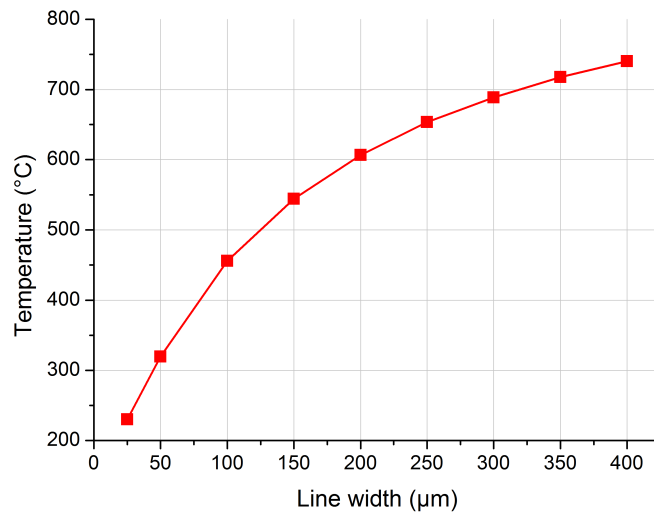


Figure 3.4: Sintering temperature as a function of conductor line width.

Figure 3.4 shows that thinner lines achieve a significantly lower temperature than wider lines if the same sintering parameters are used. This is because

the heat dissipation to the substrate material is most effective closer to the edges of the pattern due to horizontal thermal conductivity in addition to the vertical conductivity as illustrated in Figure 3.5. Figure 3.5 show thermal distributions of 50 and 400 μm wide conductor lines in a shared structure.

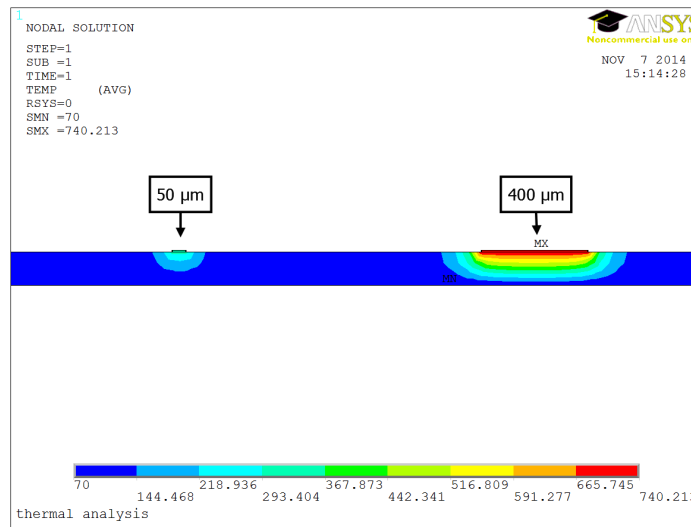


Figure 3.5: FEM simulated temperature of 50 μm wide (on left) and 400 μm wide (on right) tracks on top of 125 μm thick polyimide foil.

Modeling presented in Figures 3.4 and 3.5 is based on the laser sintering model but similar behavior is present in any sintering method which only presents energy flow on the top face of the printed structure. This includes also plasma and IPL sintering. Sintering methods relying on isotropical heating, such as conventional thermal sintering in a convection oven, do not have the same issue.

3.2.3 Intense pulsed light sintering

IPL sintering utilizes a high intensity wide spectrum lamp to quickly heat the inkjet-printed structure up to the sintering temperatures. [82] High intensity of the lamps enables very rapid sintering of metallic nanoparticles and is thus considered a primary candidate for R2R-fabrication of printed electronics. [6, 82, 83]

Figure 3.6 illustrates the basic principle of an R2R IPL sintering unit. The high intensity lamp, or in the case of wider fixtures the series of high intensity lamps, remains in a fixed position as the substrate containing the printed patterns is transported underneath, thus being exposed to the light. IPL sintering enables very high fabrication volumes, single pass sintering can be done for substrates up to 2 m in width and processing speeds up to 100 m/min can be used. [84] This enables a production volume of 200 m²/min. At these speeds the production capacity will be limited by printing techniques as well as substrate handling after sintering instead of the sintering process.

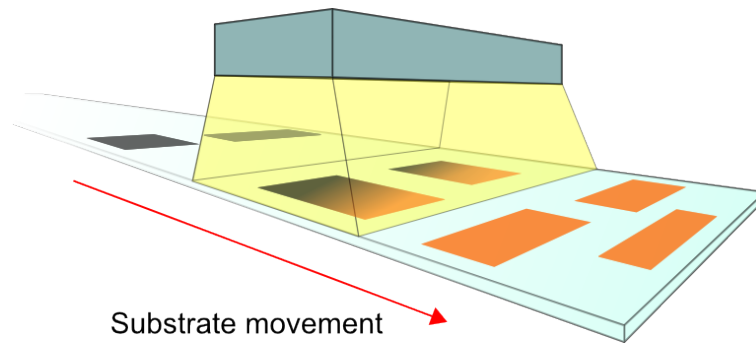


Figure 3.6: Illustration of the basic functionality of an R2R IPL sintering unit.

Most polymer substrates are practically invisible for the spectrum used by IPL sintering equipment (350-900 nm) so radiation is not absorbed by the substrate material. Most of the inkjet-printed nanoparticle inks are dark in color before sintering and therefore they absorb the energy from the photonic radiation with good efficiency. Thus the sintering process only heats up the printed structure and not the substrate material. In theory this enables the use of low temperature substrate materials but the secondary heat conducted from the printed structure may be high enough to cause serious thermal damage to the substrate materials even though the substrate itself does not absorb the light directly. [P2] [73]

3.2.4 Other sintering techniques

Other sintering techniques are studied as well but are not covered in the research related to this thesis. The most prominent ones are electrical and chemical sintering.

Electrical sintering relies on electric field or electromagnetic radiation (EMR) inducing a current running through the printed pattern and sintering the structure through Joule heating. Electric sintering can be divided into different sub-categories based on the method of effect, electric field/EMR, or on the used frequency. Electrical sintering can be done both with a direct or alternating current whereas EMR-based sintering is usually done in microwave wavelengths. [48,72]

The disadvantage of both these techniques is the requirement of pre-existing electrical conductivity to start with to enable the coupling of the electromagnetic radiation and transfer of the current through the structure. This means that basically these sintering techniques must be combined together with other sintering techniques which provide the initial conductivity. [48,85]

Electrical sintering is a rapid sintering technique, partly because the nature of Joule heating and sintering of the printed structure means that there is very strong positive feedback as heating the metal nanoparticle ink decreases its resistance and thus increases the power of the Joule heating. Therefore a very fast response thermal control is necessary to limit the sintering power.

Electrical sintering is, however, highly dependent on the pattern geometry as the absorbed sintering power depends on the absorbing efficiency and is dictated by conductivity and RF matching between the pattern and the energy source. [48,72] Therefore device architecture including both large areas and fine features is likely to be unevenly sintered. The effect is the same as modeled for laser sintering in the section 3.2.2 and illustrated in Figures 3.4 and 3.5, although the root cause is different. Also the sintering of metal nanoparticle ink changes the resistance and therefore the impedance matching between the radiating power source and the printed structure to be sintered.

Electrical sintering may also have unpredictable side effects when used to sinter printed structures which are part of hybrid designs and when the circuitry also includes ICs. Strong electric fields and EMR fields may damage ICs and cause logical and functional errors which are hard to identify in quality testing. This effect can be reduced by varying the EMR frequency and thus preventing the formation of standing waves inside the IC.

Chemical sintering has also been studied and it relies on decomposition of the protective polymer layer with a chemical which is either deposited on top of printed ink or precoated on the substrate. [86,87] Decent conductivity has

been reached with chemical sintering but the mechanical performance has not been discussed.

Research has also been done into combining several sintering methods into one process. [71] The idea is to combine the benefits and advantages of several different techniques while negating the disadvantages.

One example is combination of IPL and electrical sintering. IPL sintering is a very effective process but the efficiency drops as the printed metal structure is sintered further and the surface becomes more reflective for light wavelengths used by the IPL sintering unit. Electric sintering, on the other hand, is a slow process to begin with as it requires an initial electrical conductivity for electromagnetic coupling and the efficiency is increased as the sintering progresses and the conductivity increases. Therefore the two sintering methods can be used to supplement each other.

3.3 Comparison

In research and publications related to this thesis plasma, laser and IPL sintering were analyzed and compared to traditional thermal sintering based on their electrical and mechanical performance. The main results of the research are listed in P3, while plasma sintering is covered individually in P1, laser sintering in P4 and P5 and IPL sintering in P2 and P5.

The main conclusion of the studies was that IPL sintering was identified as the most promising sintering technique for silver nanoparticle inks [P3] enabling both high performance and large-scale fabrication. For copper nanoparticle ink both laser and IPL sintering were deemed highly feasible. The two sintering techniques are, however, identified as complementary, not exclusive to each other as they have very different strengths and advantages. IPL sintering excels in rapid large scale fabrication whereas laser sintering is more flexible and benefits from a high level of process control and is therefore better suited for smaller scale applications, or applications requiring high level of customization and precision.

Plasma sintering is a deemed usable technique but only on a limited range of applications due to low conductivity and poor mechanical performance. The low processing temperature of the plasma sintering is, however, a significant

benefit as it enables the use of low cost substrates. However, only low pressure argon plasma sintering was studied in research related to this thesis and the atmospheric plasma sintering may have better performance and thus wider range of feasible applications. [75]

FEM modeling of the laser sintering process is presented in publication P4, and the main observation of the study was that the sintering temperature alone cannot explain all the sintering behavior of the copper nanoparticle ink even though it clearly plays a significant part in triggering the sintering effects. In publication P5, IPL and laser sintering are compared with the main focus on energy density per area during sintering. The main conclusion is that energy density alone is not an explaining factor for sintering performance, at least if different sintering methods are compared to each other. The conclusion of these two findings is that the sintering process is a complicated process and neither the sintering temperature nor the sintering energy density alone can be used to assess the sintering performance even though they give certain data to begin with.

3.3.1 Microstructure

The microstructure of inkjet-printed and sintered patterns is an essential part in understanding the progress of the sintering process and the effects that dictate the electrical and mechanical performance. Microstructure analysis has been included in publications P2, P3 and P5.

The microstructure of nanoparticle silver ink has been covered in publications P2 and P3. Publication P3 includes comparative analysis of electrical and mechanical performance as well as microstructural observations between different sintering techniques. Figure 3.7 presents the SEM images of microstructures for thermal (3.7a), IPL (3.7b), plasma (3.7c) and laser (3.7d) sintered silver (Ag) ink.

Figures 3.7a and 3.7b show cross-sectional SEM images of thermally and IPL-sintered Ag-ink structures. The images clearly show that both sintering methods enable very uniform and high density microstructure, which clearly contributes also to the high conductivity achieved with these techniques.

Figure 3.7c clearly shows the top-to-bottom sintering behavior of the plasma-sintering, which has been reported earlier by Reinhold et al. [70] Sintering is

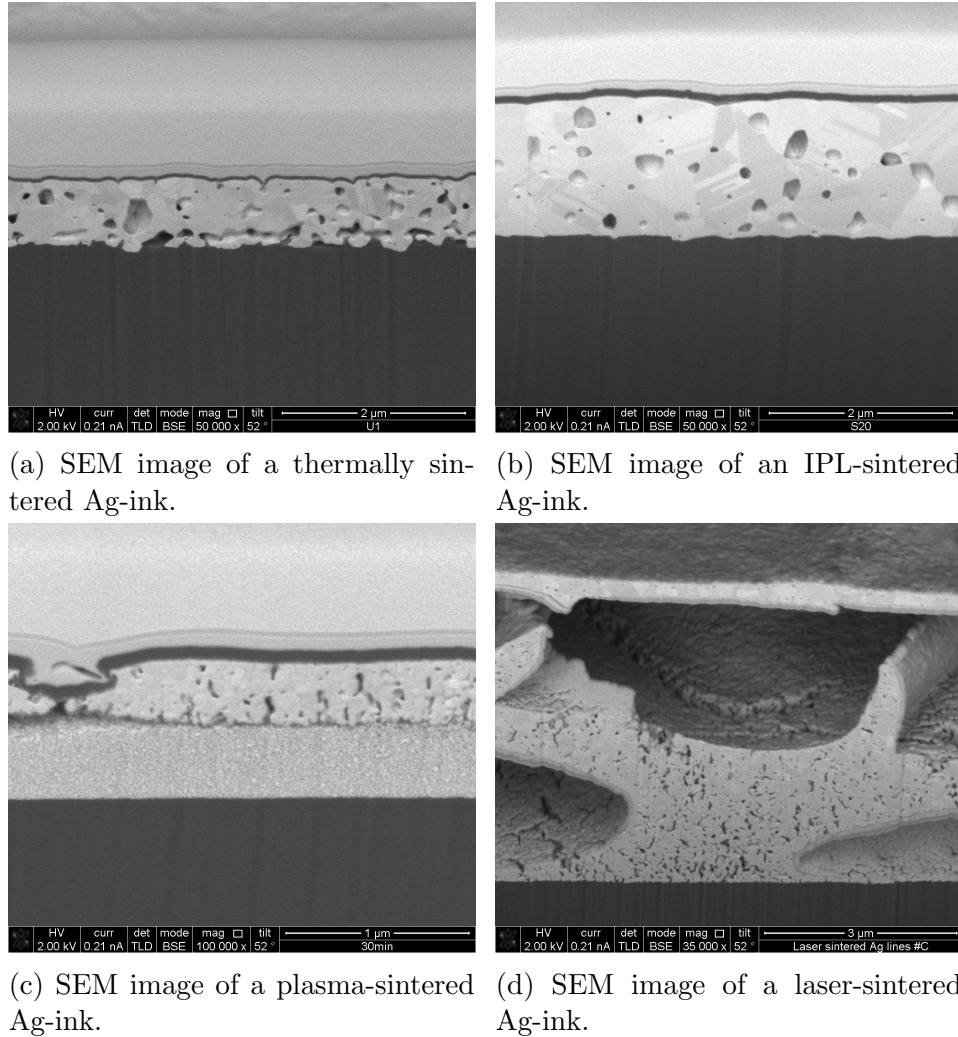


Figure 3.7: SEM images of different sintering techniques on Ag-ink.

seen to progress downwards as the sintering time is increased. The top-to-bottom sintering behavior is thought to be one of the main reasons for poor adhesion observed between the plasma-sintered structures and the substrate in publications P1 and P3.

Figure 3.7d shows a cross-sectional SEM image of a laser-sintered Ag-ink pattern. The microstructure of laser-sintered Ag-ink is found to be highly non-uniform and large voids are present. This is thought to be because of very rapid sintering by laser-heating and therefore rapid evaporation of solvent remains. Decent conductivity was achieved with laser-sintering but obviously the poor microstructure hampers both electrical and mechanical

performance. Similar behavior was not observed with IPL sintering despite it being very rapid process as well. The main factor is thought to be the higher processing temperature of the laser-sintering which causes more violent solvent evaporation.

Research on sintering of copper (Cu) nanoparticle inks is reported in publications P4 and P5. Cross-sectional SEM images of the microstructure of IPL and laser-sintered patterns are presented in Figure 3.8, IPL in 3.8a and laser in 3.8b.

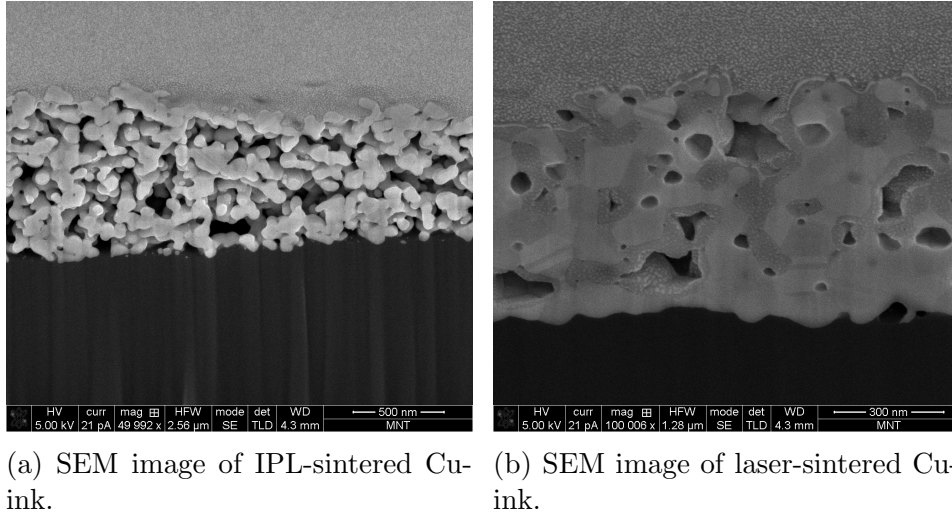


Figure 3.8: SEM images of different sintering techniques on Cu-ink. [P5]

Laser-sintering of Cu-ink, see Figure 3.8b, provides a significantly different microstructure than with Ag-ink, see Figure 3.7d. The microstructure of laser-sintered Cu-ink does not have similar voids and non-uniformities as seen in Ag-ink. The differences in ink composition, mostly different solvent and dispersion materials, are the main reason for the differences observed between the two ink materials.

3.3.2 Adhesion

In addition to the electrical performance also the mechanical performance of the inkjet-printed structures is assessed. The mechanical performance is evaluated based on the formed adhesion between the printed structure and

32 Sintering of metal nanoparticles for internal interconnections

the substrate during the sintering. Adhesion is tested using the ASTM D-3359-09e2 peel-off test standard using adhesive tape. [88]

In the ASTM D-3359-09e2 peel-off adhesion test the specified test pattern, a 15 mm by 15 mm square, is divided into 49 smaller test squares with a specific tool. A peel-off test is done on the scratched pattern with the standardized adhesive test and the adhesion is evaluated based on the structure remaining on the substrate after the tape peel-off. The ASTM D-3359-09e2 test does not give absolute values for adhesion but gives qualitative results and enables a simple comparative study. This makes it possible to analyze the effects of different sintering treatments on the adhesion. The ASTM D-3359-09e2 test procedure is explained in detail in publication P1.

Adhesion testing is covered in publications P1, P2 and P3. The conclusion of the research reported in the related publications is that the achieved adhesion between the printed structures and the substrate material is highly dependent on the used sintering method and parameters. For Ag-nanoparticle inks IPL and thermal sintering enabled the best adhesion whereas laser and plasma sintering failed to produce adequate adhesion for microelectronics applications. Adhesion studies on Cu-nanoparticle inks have not been published but very good adhesion has been observed with both laser and IPL sintering.

Figure 3.9 presents the peel-off adhesion test results for IPL-sintered structures printed with Ag-nanoparticle ink. [P2] The top image shows the test structure as-sintered, before the peel-off adhesion test and the bottom-left image shows the test structure after the peel-off adhesion test. The bottom-right image shows the adhesion tape used in the peel-off tape and the material removed from the structure during the peel-off test. Figure 3.9 shows minimal peeling of the printed structure and the results belong either in the 4B or 5B categories, the highest categories in the ASTM D-3359-09e2 standard.

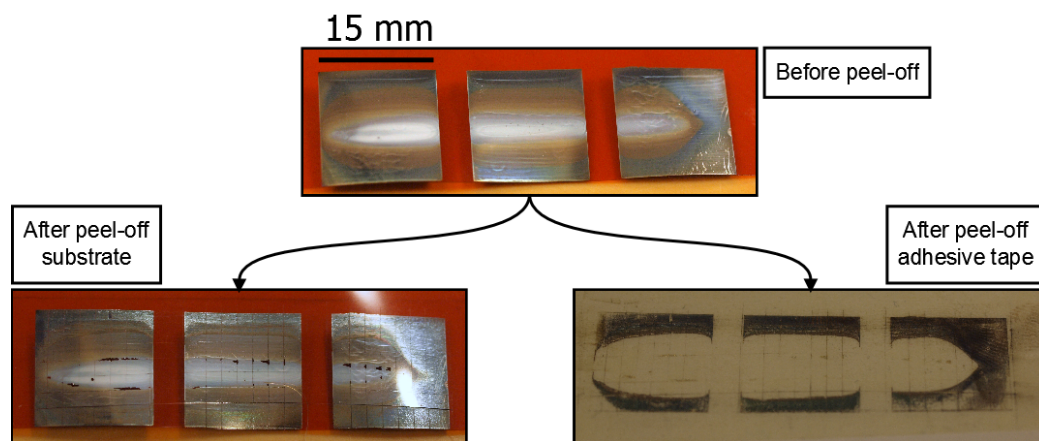


Figure 3.9: Peel-off adhesion test results for IPL-sintered Ag-nanoparticle ink. The size of each test pattern is 15 mm by 15 mm. [P3]

Chapter 4

External interconnections

Advances in the electronics industry are heavily dictated by customer demand. As customers are used to a certain level of functionality the change of manufacturing technique can not mean a drawback in functionality as customers are reluctant to take "a step backwards" in terms of functionality and features.

Developers of printing techniques dream of all-printed electrical devices and some progress has already been made in very simple devices. [12, 89, 90] All-printed structures and applications have, however, the major drawback that materials and processes heavily limit their potential functionality. Functionality is especially limited by the low performance of printable semiconductive materials. [26] Consequently, the level of functionality required of today's consumer electronic devices can be achieved only by using a hybrid technique that incorporates printed electronics with traditional silicon based CMOS components and takes advantage of the strengths of both techniques. [14, 18] Inkjet-printing can be used in electronics miniaturization and integration to create new kinds of component connections. [17, 24] A hybrid technique poses some challenges for component interconnections as well. The main issue is the component attachment to an inkjet-printed circuit board.

Requirements for external interconnections include electrical and mechanical performance as well as long-term reliability. Requirements, especially long-term reliability related ones, are highly dependent on the designated use case scenario and the environmental stresses the device is designed to face during its lifetime.

4.1 Conductive adhesives

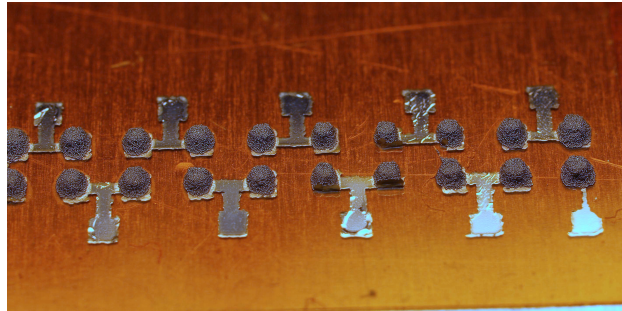
Soldering is by far the most widely used component attachment method in today's electronics industry, but it cannot be used with inkjet-printed silver structures without proper plating of the component pads. This is caused by the material incompatibilities between inkjet silver ink and solder caused by the leaching effect of the traditional solder material (SnAgCu). [91]

In traditional electronics, leaching is solved by adding silver to the solder material, which though not canceling the effect obviates it as a problem in traditional thick film manufacturing. In inkjet-printing, however, the layer thickness is so much smaller that even the slightest leaching can cause open circuits and thus functional errors. [92, 93]

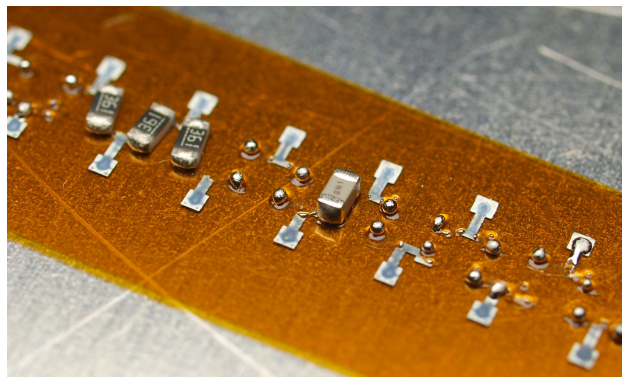
Figure 4.1 shows the leaching effect on inkjet-printed PCB fabricated with silver nanoparticle ink. Figure 4.1a shows the PCB and the stencil-printed solder paste on component pads before the reflow-process. Figure 4.1b shows the PCB after the reflow-process as the solder material has leached the silver conductor. Open circuits are clearly visible on the picture next to each component pad.

Solder also has a similar leaching behavior to printed copper structures but the phenomenon is not as strong as with silver and does not prevent the use of solder with printed copper. Solder processing suffers from poor wetting of the solder material on top of the printed copper due to high surface roughness and heterogeneity. [91] Optimization of copper printing and post-processing might help reduce the incompatibility problems. Solder processing would be an important step in increasing the compatibility between inkjet-printing and traditional electronics manufacturing processing and therefore also very important in making inkjet-printing more likely to be introduced into large scale electronics manufacturing.

Adhesives do not have the same leaching effect as solders and are therefore more compatible with inkjet-printing materials. Conductive adhesives also have several other advantages over solder materials. ECAs have lower processing temperatures than solder materials and therefore they enable the use of cheaper substrates and their high elasticity makes them preferred choices for flexible applications. [94]



(a) Solder material on inkjet-printed patterns before reflow-process.



(b) Solder material on inkjet-printed patterns after reflow-process.

Figure 4.1: Leaching of inkjet-printed silver nanoparticle ink on solder material.

ECAs have also some drawbacks compared to solder materials, such as the tendency to contact resistance shift during exposure to elevated temperature and humidity as well as poor impact resistance. [94] Adhesive interconnections are also significantly harder to rework than soldered ones, which makes replacing faulty components in electronics assemblies more difficult and more expensive. Adhesive materials also lack the self-alignment capabilities which help solder interconnections to get rid of minor misalignment or misplacement errors.

Usability of ECAs as replacements for solder connections has been investigated in flexible electronics in general. [95,96] The conclusion of these studies has been that adhesives do not have as good reliability as solder interconnections but are still adequate for all but the most demanding applications. It has also been noted that failure mechanisms may be different for the different

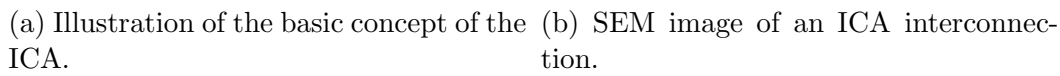
interconnection materials: in solder interconnections cracking usually occurs through the solder material itself whereas in adhesive connections failure is due to a delamination in the interface between the adhesive and a component terminal or cracking of the component lead. Stress relaxation of the adhesive base material may also cause problems in adhesive connections. [96] Stress relaxation also makes adhesive materials challenging subjects for reliability studies as they make failures highly reversible after stresses have been removed. On the other hand this feature makes them well suited for actual applications.

The electrical performance of the ICA attachment is deemed adequate for SMD attachment and found to be a viable solution for component attachment on inkjet-printed substrates. [P6-P8] However, because electronic applications must sustain long-term use, performance alone is not enough. Therefore, ICA connections also need to be tested and analysed for long-term reliability in order to evaluate their overall feasibility on inkjet-printed applications.

4.1.1 Isotropically conductive

ICAs are electrically conductive adhesive materials which conduct electricity equally in all directions, thus the name "isotropic". ICAs work basically exactly the same way as traditional solder materials and are therefore the most straightforward replacements. ICA material consists of a matrix material, usually epoxy, acryl or silicone, and a filler material which provides the electrical conductivity. Isotropic conductivity is achieved as the conductive filler content of the ICA material is above the percolation threshold and therefore conductive paths are formed in all directions. [97] In most cases the filler material is metal, most commonly silver flakes. Figure 4.2 illustrates the structure of an ICA interconnection.

Figure 4.2a shows the basic concept of the interconnection and is not to scale. A SEM image of an ICA interconnection is presented in Figure 4.2b. The SEM image is from a flex-on-flex structure in which two inkjet-printed flexible PCBs are attached to each other using ICA interconnection. The structure is the same as described in P8. Substrates (polyimide films), printed silver ink layers as well as conductive filler particles of the ICA material are clearly visible in the SEM image. Thickness of the printed silver layer is about 3 μm and thickness of the ICA layer is about 20 μm .

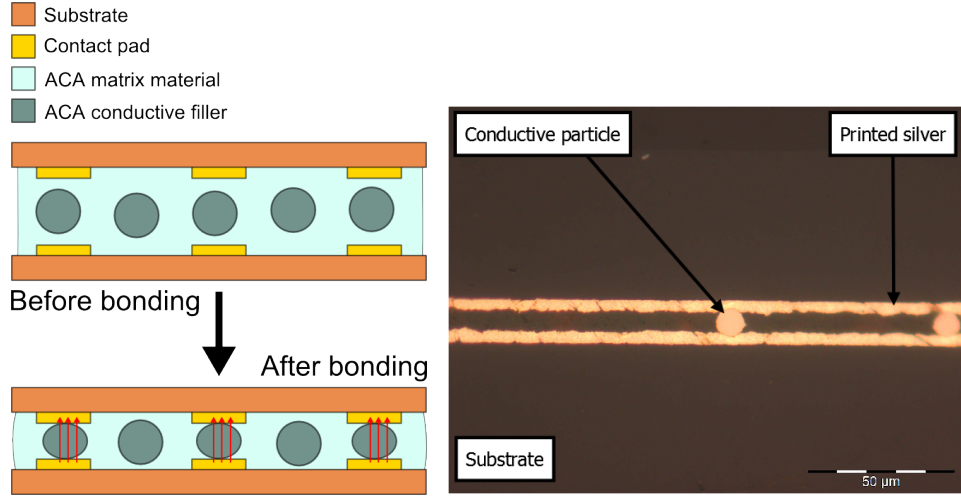


ICA material is deposited, usually with stencil-printing or dispensing, on the component pads and the electricity can flow through the ICA material. As ICA conducts electricity in all directions, the depositing of the ICA material has to be done so that no short circuits are formed between component pads. Therefore deposition accuracy limits the feasible feature sizes for ICA process. After depositing and component placement the ICA material needs to be cured, which is usually done by heating.

ACA materials, unlike their ICA counterparts, are by default non-conductive. ACA material consists of a basic matrix material, which provides mechanical support, and electrically conductive particles, which provide electrical conductivity. ACA material is not conductive in its default form as the conductive filler content of the ACA material is below the percolation threshold and thus conductive paths are not formed through the ACA structure. [97]

Functionality of the ACA material is based on the bonding process in which temperature and pressure is applied to the interconnection area. [98, 99] As the interconnection thickness decreases due to the pressure, the percolation threshold of the conductive filler material is locally and unidirectionally achieved and electrical conductivity is reached. The bonding process also includes heating which cures the ACA matrix material making the interconnection mechanically stable.

The cross-sectional structure of the ACA interconnection is presented in Figure 4.3. Figure 4.3a illustrates the basic structure of the ACA interconnection and shows how application of pressure during the bonding process locally increases the conductive filler content above the percolation threshold. The figure is not to scale. Figure 4.3b presents an optical microscope image of an ACA interconnection between two inkjet-printed flexible PCBs. The structure is the same as presented in publication P8. Silver conductor lines as well as the conductive filler particle can clearly be seen in the image. Figure 4.4 shows a SEM image of an interface between a conductive filler particle, gold-coated nickel particle and inkjet-printed silver trace.



(a) Illustration of the basic concept of the ACA interconnection. (b) Optical microscope image of an ACA interconnection.

Figure 4.3: ACA interconnection illustrations.

As the ACA material is not conductive in its default form and only forms electrical connections on interconnection direction, the ACA material can be applied on a larger area without a risk of short circuits between component pads. This is illustrated in Figure 4.3a and can also be seen in Figure 3 on P8.

4.2 Other methods

The accuracy of inkjet-printing is high enough to enable a direct connection of a bare silicon chip with grid array connectors. [14, 17, 24] The use of printed

interconnections enables the use of bare silicon chips thus saving process steps in component manufacturing and space in both area and height in final assembly. [18]

Direct component connection makes it possible to fabricate circuit patterning and the component attachment on the same process step, which makes it possible for the direct printed component connections to partially replace wirebonding in the component assembly. Tighter packaging solutions would be greatly beneficial in today's electronics manufacturing, especially when considering mobile devices and their size requirements.

Wirebonding on inkjet-printed structures has also been tested. [100] However, due to mechanical properties, mainly hardness and low Young's modulus, of the printed structure wirebonding directly on the printed surface is extremely challenging. Additional coating is needed to improve the wirebondability of the printed surface.

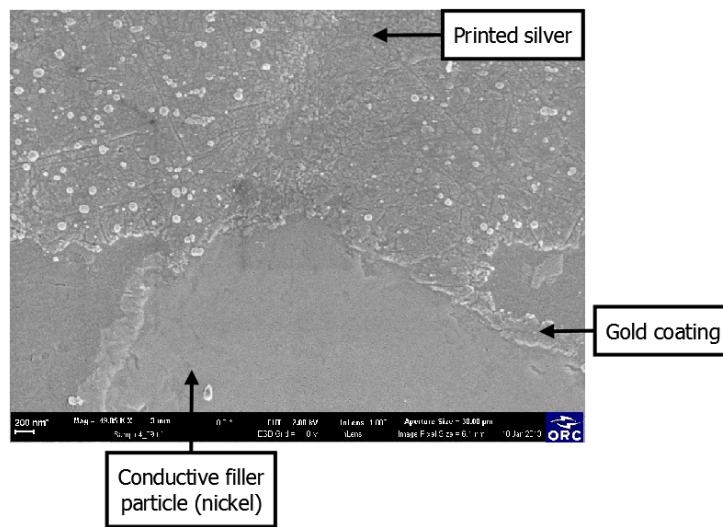


Figure 4.4: SEM image of an interface between a conductive filler particle of ACA material and inkjet-printed structure.

4.3 Reliability

It is not enough for an electric device to work right from the manufacturing line but it also has to keep working as intended continuously for its entire lifetime. Intended lifetimes of electric devices range from a few years for consumer electronic applications (or even days in case of short term single use devices which are discarded after use) to decades for aviation and space application. [101] As the intended lifetimes and the use scenarios and environments of different electrical devices vary greatly, so do their reliability requirements. Therefore reliability assessment has to be done specifically for each device type.

In most cases reliability problems are caused by external factors: extreme temperatures, rapid temperature changes, high humidity, salt (or other corrosive pollutant) or mechanical stresses such as shocks and vibrations. [101] Sometime the problem can also be because of internal causes such as material degeneration due to aging or thermal stress generated by the operation of the device itself. No device can be fully protected against all the stresses and therefore understanding the case specific main stresses is important in order to better protect the device against specific stresses.

Reliability testing is challenging as the long-term reliability of devices can not be tested in full length as this would mean a several year delay in product launch. Therefore long-term reliability of electronic devices is done by using accelerated stress tests. The idea is that the devices are exposed to elevated environmental stresses so that they will face a similar amount of stress than the device is estimated to face during its supposed lifetime in normal use conditions in a shorter time. This shortens the required test time from years to weeks.

Environmental stress acceleration has to be done carefully, though, as the acceleration may change the failure mechanism of the tested device. For the accuracy of the analysis it is important that the failure mechanism and mode are the same as the device faces in its supposed lifetime in normal use conditions. Acceleration factors can be calculated for accelerated stress tests so that equivalent lifetime can be predicted based on the stress factors and survival time in elevated conditions. [101]

4.3.1 Experimental testing

Reliability of printed electronics itself has been studied and inkjet-printing itself has not been observed to cause significant reliability issues as long as environmental protection is taken care of. [61, 102, 103] However, no research had been done on the reliability of component interconnections on inkjet-printed PCBs prior to this thesis work. Reliability research related to this thesis is focused on the long-term reliability of component interconnections on inkjet-printed PCBs on temperature cycling as well as elevated temperature and humidity conditions.

Temperature cycling

In a temperature cycling stress test the temperature of the test chamber is continuously cycled between set extremes. The effect of the stress test is based on the mechanical stresses caused by the different coefficients of thermal expansion (CTE) of different materials in the interconnection assembly. [104]

Temperature cycling of ICA interconnections was done in temperatures between -40 and 125 °C in a thermal cycling chamber with continuous resistance logging for fault detection. The measurement setup is explained in detail in publication P6 and P7. The main observation of the temperature cycling of the ICA interconnections is that using inkjet-printing to fabricate the PCB has no effect on the reliability of the interconnections.

A comparison was made between ICA-based interconnections on inkjet-printed and lithography-fabricated PCBs and no significant difference was observed. Similar fault mechanisms and failure locations were observed in both cases. An additional comparison was made between ICA-based and solder interconnections. Solder was found to be superior and the difference is contributed to the superior mechanical performance of the solder compared to the ICA material.

As the main task of the thesis is to evaluate different interconnection techniques for printed electronics applications it can be concluded that the ICA-based interconnections, while not as reliable as solder-based ones, are a feasible method for connecting external components on inkjet-printed PCBs. An

important observation is also that the reliability of the assembly is mainly affected by the used interconnection material and the used PCB-fabrication method was not observed to have any effect.

The failure modes and mechanisms observed in P7 are similar as earlier reported by Kuusiluoma et al. and therefore it is also assumed that the used acceleration was appropriate. [96] In general the findings of the thermal cycling match earlier publications. [P7] [105] Figure 4.5 presents a SEM image of a failed ICA-interconnection after a thermal cycling stress test. The image shows a crack propagating through the plating of the SMD component.

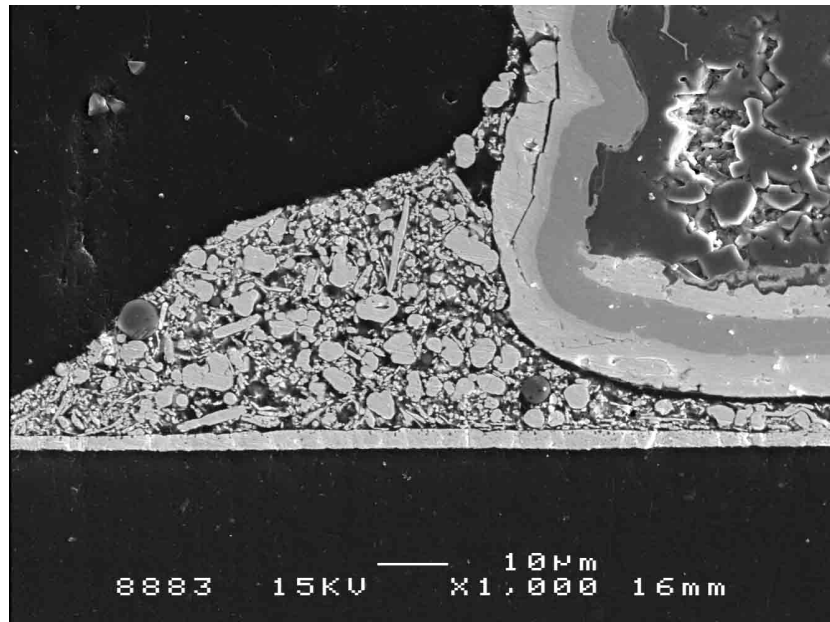


Figure 4.5: SEM image of an ICA interconnection after failing in a thermal cycling test. [P7]

Elevated temperature and humidity

Elevated temperature and humidity conditions are used to accelerate high humidity related phenomena such as moisture absorption. Moisture absorption is an issue in electronics devices as polymer materials, which are commonly used as substrate and packaging materials in electronic assemblies, absorb moisture, which leads to material expansion and therefore mechanical stresses in interconnections. One of the most commonly used test condition is

"85C/85RH" which means an 85 °C temperature and 85 % relative humidity but harsher conditions are used as well to further accelerate the testing. [106]

For this thesis work, long-term reliability of both ICA and ACA-based interconnections were tested under elevated temperature and humidity conditions and the effect of using printed electronics was analyzed. ICA and ACA were used for connecting two flexible inkjet-printed PCBs together. PCBs printed with silver nanoparticle ink on polyimide substrate were tested in a "85C/85RH" test and the contact resistance was monitored using a continuous resistance measurement in order to identify the failure times. Both contact methods were found to be feasible but ACA was observed to enable lower contact resistance as well as better long-term reliability. The research setup and the results are explained in detail in publication P8.

The elevated temperature and humidity condition accelerates the moisture absorption of the adhesive matrix material and causes swelling on the assembly. The swelling in turn causes mechanical stresses in the adhesive material and results in cracking inside the assembly. Figure 4.6 presents a SEM image of a failure in an ICA interconnection under elevated temperature and humidity conditions. The cracking of the adhesive matrix material interrupts the conductive path between the connector pads and causes an open circuit.

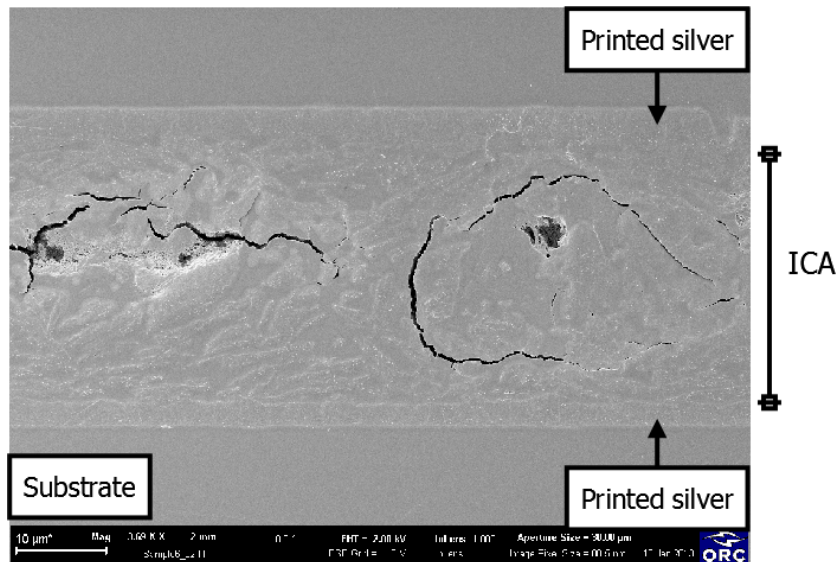


Figure 4.6: SEM image of an ICA interconnection after failing in an "85C/85RH"-test.

In the reliability testing it was observed that, in addition to cracking inside the adhesive matrix material, the failures occurred in the interfaces between the adhesive material and the inkjet-printed structure. This shows that inkjet-printed traces have good adhesion on the substrate. Adhesion between the printed structure and the adhesive material should be optimized by altering printing and sintering process parameters of the printed structure as well as those of the adhesive process.

Decent long-term reliability was achieved with all the tested materials, and acryl-based ACA provided very good results. It is not clear if the interface delamination is because of the use of inkjet-printing for PCB fabrication, but no obvious disadvantage comes from using inkjet-printing.

Chapter 5

Conclusions

As printed electronics, including inkjet-printed electronics, gains popularity and starts moving from research laboratories into electronics manufacturing more information is needed on the fabrication process on a larger, system-level, scale. This thesis and the related studies and publications look into the sintering of inkjet-printed nanoparticle inks and forming external interconnections to printed structures.

The objective of the thesis was to improve the understanding of different interconnection fabrication techniques for inkjet-printed electronics. The main hypothesis was that a feasible and reliable method for fabricating interconnections on inkjet-printed structures can be developed, and this has been confirmed in the publications related to this thesis. The thesis has increased the knowledge on fabrication of both internal interconnections (alternative sintering methods) as well as external connections to inkjet-printed structures.

Sintering of nanoparticle inks is an important step in inkjet-printing and better understanding of the sintering process and the thermal phenomena during the sintering enables use of a larger variety of materials. Several different sintering techniques are studied in the work related to this thesis and their usability for inkjet-printed nanoparticle inks have been evaluated as there is a strong need to replace traditional thermal sintering with a faster and more efficient technique.

IPL- and laser-sintering were found to be the most promising alternative techniques due to their good conductivity and adhesion as well as short processing times. IPL-sintering is the fastest of the tested alternative sintering techniques and very well suited for large scale electronics manufacturing. Laser-sintering is a slower technique but the digital control enables a high level of customization and superior process control over IPL. Digital location and optical power controls make it possible to accurately control the laser power according to pattern geometry and prevent unwanted thermal loading on sensitive components and materials.

Furthermore, external interconnections are needed as the printed structures cannot provide the complex logical functionality required of modern electronic devices and thus traditional CMOS-components are needed in addition to printed structures. External interconnections are also needed to provide the printed devices with data and power connections to the outside world through the next integration level.

As the traditional soldering technique is not compatible with inkjet-printing due to material incompatibilities, alternative interconnection methods were studied. Electrically conductive adhesive materials were evaluated and tested for both component interconnections as well as circuit board to circuit board interconnections and they were deemed suitable options. Although neither the electrical performance nor the long-term reliability of the adhesives were as good as those of the solder-based interconnections, the difference was deemed to be due to the material difference between adhesive and solder, not the fabrication method used for circuitry.

Publication 1, entitled "Conductivity and Adhesion Study of Plasma-sintered Nanoparticle Silver Ink", studied the usability of low pressure plasma-sintering for the sintering of inkjet-printed nanoparticle silver inks. Plasma-sintering was found to enable feasible electrical conductivity for low performance electronics (~ 10 % of bulk silver). Adhesion between the printed layer and used polymer substrate was, however, found to be poor and ill-suited for most microelectronics applications.

Publication 2, entitled "Comparison of photonic sintering of two inkjet-printed nanoparticle silver inks", sought the differences in photonic sintering behavior between two inkjet-printed nanoparticle silver inks. Excellent adhesion was achieved between the printed structure and the substrate with both inks. Also the electrical conductivity was good with both inks and no significant differences were observed. However, the microstructure analysis

revealed significant differences between the two inks and it was concluded that the microstructure cannot be used by itself alone as an indicator of the electrical conductivity.

Publication 3, entitled "Alternative Sintering Methods Compared to Conventional Thermal Sintering for Inkjet Printed Silver Nanoparticle Ink", compares IPL-, laser- and plasma-sintering as alternatives to thermal sintering of inkjet-printed silver nanoparticles. The novelty of this publication was the comparison of different sintering techniques under similar conditions. All the sintering techniques investigated in the paper are studied and reported earlier, but not under the same conditions. Testing the different techniques under the same conditions is important as it helps get rid of testing condition related effects on the sintering performance. IPL-sintering was found to be the most promising of the alternative sintering techniques as it enables good electrical conductivity and mechanical performance in a very short processing time. Decent electrical conductivity was also achieved with laser- and plasma-sintering but both techniques produced poor adhesion between the printed structure and the polymer film used as a substrate.

Publication 4, entitled "Characterisation of Laser Sintering of Copper Nanoparticle Ink by FEM and Experimental Testing", studied the laser-sintering of inkjet-printed copper nanoparticle ink and compared the experimental electrical conductivity results and simulated thermal data. The results showed that the achievable conductivity of the printed structures cannot be predicted directly from the simulated sintering temperature as there are other factors affecting the sintering process as well. Sintering temperature can, however, be used for rough evaluation of the sintering performance.

Publication 5, entitled "Comparison of laser and intense pulsed light sintering (IPL) for inkjet-printed copper nanoparticle layers", compared laser- and IPL-sintering of inkjet-printed nanoparticle copper ink based on the achieved conductivity and the microstructure after sintering. Both sintering techniques were found to enable good conductivity (over 20% of bulk copper conductivity) and to be suitable alternatives for sintering of copper nanoparticle inks. The scale of the processability, however, differs significantly as IPL is well suited for processing of large areas very quickly whereas laser is more suitable for highly adaptive processing of small features. Therefore the two techniques can be seen as complementary techniques instead of competitive ones. Cracking phenomenon was observed with both sintering techniques even though the behavior differs. Further research is needed to better understand the cracking with both techniques.

Publication 6, entitled "Characterization of ICA-Attachment of SMD on Inkjet-Printed Substrates", evaluated the electrical performance of ICA-based component interconnections on inkjet-printed structures. ICA-based interconnections were found to be inferior to solder-based ones but the difference is shown to be caused by the ICA material itself and not the use of inkjet-printed electronics. The electrical performance of the ICA-interconnections was, however, found to be suitable for most electronics applications and design rules were discussed.

Publication 7, entitled "Reliability of ICA attachment of SMDs on inkjet-printed substrates", continued the study of ICA-based component interconnections and evaluated the long-term reliability of the interconnections based on the accelerated stress testing results. The results showed that solder-based interconnections had better long-term reliability but no difference was observed between ICA-based interconnections on inkjet-printed and photolithography-fabricated circuit boards. Based on the combined results of publications 6 and 7 it was concluded that using inkjet-printing for circuitry fabrication does not affect neither the electrical performance nor the long-term reliability of the assemblies and ICA was deemed a suitable interconnection method for inkjet-printed electronics. Further studies are, however, needed for interconnection techniques to form more generalized knowledge on effects of using inkjet-printing, or other printing techniques.

Publication 8, entitled "Reliability of Flex-to-Flex Interconnections on Inkjet-Printed PCBs Electrically Conductive Adhesives", studied connecting two inkjet-printed circuit boards together with ACA- and ICA-based interconnections. Both methods were found to be suitable but ACA was found to enable lower contact resistance and better long-term reliability under accelerated stress testing.

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Publication 1

Conductivity and Adhesion Study of Plasma-sintered Nanoparticle Silver Ink
J. Niittynen, E. Halonen, M. Mäntysalo, J. Perelaer, U.S. Schubert and
D. Lupo

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Publication 2

Comparison of photonic sintering of two inkjet-printed nanoparticle silver inks

J. Niittynen, M. Mäntysalo, D. Lupo, R. Abbel, J. Perelaer and U.S. Schubert
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Publication 3

Alternative Sintering Methods Compared to Conventional Thermal Sintering
for Inkjet Printed Silver Nanoparticle Ink

J. Niittynen, R. Abbel, M. Mäntysalo, J. Perelaer, U.S. Schubert and D. Lupo
Thin Solid Films, 2014, 556, 452-459.

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Publication 4

Characterisation of Laser Sintering of Copper Nanoparticle Ink by FEM and Experimental Testing

J. Niittynen and M. Mäntysalo

IEEE Transactions on Components, Packaging and Manufacturing Technology, 2014, 4, 2018-2025.

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Publication 5

Comparison of laser and intense pulsed light sintering (IPL) for inkjet-printed copper nanoparticle layers

J. Niittynen, E. Sowade, H. Kang, R.R. Baumann and M. Mäntysalo

Scientific Reports, 2015, 5, 8832.

Publication 6

Characterization of ICA-Attachment of SMD on Inkjet-Printed Substrates

J. Niittynen, V. Pekkanen and M. Mäntysalo

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Las Vegas, Nevada, USA, 2010.

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Publication 7

Reliability of ICA attachment of SMDs on inkjet-printed substrates

J. Niittynen, J. Kiilunen, J. Putaala, V. Pekkanen, M. Mäntysalo, H. Jantunen and D. Lupo

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Publication 8

Reliability of Flex-to-Flex Interconnections on Inkjet-Printed PCBs Using Electrically Conductive Adhesives

J. Niittynen, S. Koskinen, J. Kiilunen, J. Pippola, L. Frisk and M. Mäntysalo
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Errata

Figure 3, legend: Gold coating, *should read:* interface delamination

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